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COMPLETE SPECIFICATION

Improvements in a Method for Granulating Urea

We, CHEMICAL CONSTRUCTION CORPORATION, a corporation organised under the laws of the State of Delaware, United States of America, of 488 Madison Avenue, New York, 22, State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production of granulated urea. More particularly, it relates to a method for preparing storage-stable, spheroidal urea in granular form.

One of the great major problems in the commercial use of powdered urea is that it readily takes up water from the atmosphere and then solidifies into a unified mass. This difficulty has long plagued those who used urea either as an industrial commodity or as a fertilizer. Many attempts have been made to eliminate or prevent this solidification. Some of these attempts took the form of coating the individual particles of urea. Others attempted to granulate it to minimize the surface area and thus reduce the hygroscopic surface exposed to the atmosphere. Both of these measures had been successfully used on ammonium nitrate. However, it was soon found that none of the common methods of rendering compounds such as ammonium nitrate stable on storage were the least effective with urea. Thus there has not been commercially available a granulated urea that is stable on storage and retains its character of being a free-flowing granular particulate mass for an appreciable period of time.

It is the object of the present invention to supply urea in such form.

In accordance with the present invention there is provided a method of producing storage-stable, spheroidal, granulated urea which comprises heating an aqueous solution of urea to a sufficient temperature to evaporate sufficient water therefrom to produce a 90–98% urea concentration in said solution as rapidly as possible without allowing the solution temperature to exceed 100°C. for more than five hours nor to exceed 80°C. for sufficient time to produce more than 5% biuret; then rapidly raising the solution temperature to from 115°C. to 130°C., if it is not already at that temperature and spraying so-concentrated and heated solution into a gas-cooled granulation tower, whereby round discrete urea granules are obtained; collecting said granules; subjecting collected granules to the action of heated air at a temperature below the softening temperature of said granules until the residual moisture content does not exceed about 0.5% and cooling resultant granules to atmospheric temperature.

It has been found that the biuret content of the urea solution is of controlling importance in the preparation of the granula urea of the present invention. The biuret concentration must be less than about 5% if the finely granulated urea is to have desirable storage-stable properties. In order to control the biuret content, it has been found that the temperature must be controlled. For example there has previously been suggested a process of producing granulated urea according to which a urea solution is concentrated under vacuum in an evaporator, leaving the evaporator with 6% water at a temperature of 112°C., and the concentrated solution is then sprayed into an air-cooled spray drier. The granules thus formed are dried in hot air until the residual moisture content does not exceed 1%. This prior process also taught that the solution should not be maintained for any length of time above 110°C. so as to avoid biuret formation. Urea is converted to biuret at over 80°C. and the amount of biuret formed increases in proportion to increasing temperature. The

[Price 3s. 0d.]

Price 3s. 6d.

conversion of urea to biuret is not affected by water; it is solely the function of temperature and time. If the temperature is constant biuret formation proceeds at a constant rate.

In order to keep the biuret content in the solution below about 5%, the urea solution must not be at over 100°C. for longer than 5 hours. For higher temperatures the time should be shorter. It is preferable to concentrate the urea solution under as low temperature as possible, and if necessary under reduced pressure.

In order for the urea solution to give particles having the proper characteristics when subjected to granulation or spray-drying, the urea solution should be concentrated to a urea content of about 90–98% in the shortest possible period without exceeding the temperature restrictions noted above. When the urea concentration has reached about 90–98% urea, it should be swiftly heated to 115–130°C., if not already at such temperatures and immediately sprayed into a gas-cooled granulation tower from a nozzle or rotary granulator. The cooling medium in the granulation tower enters from the bottom and passes counter-current to the falling particles. The incoming cooling medium may be at room temperature. By this method the particles are solidified in perfectly round form as they fall downward to the bottom of the tower.

As long as the biuret content of the urea solution is maintained less than 5% the urea particles found on the bottom of the granulation tower will be perfectly round. However, if the biuret content of the urea solution is greater than 5% then particles will be irregular and will not have the storage-table properties that are desirable.

The granulated urea thus obtained contains about 4% moisture. If this granulated urea is stored for a long time as it is, the moisture contained will eventually cause a great deal of deterioration of the surface of the particle. The particle will then take up more water from the air and will stick to those surrounding it to form one large homogeneous mass. Therefore, the granulated urea from the granulation tower should be immediately forwarded to a further drying process.

During the supplemental drying process it is necessary to eliminate substantially all the residual moisture contained in the particles. Whatever moisture does remain after this second drying process must be evenly distributed inside the particles. If the moisture is concentrated in any particular part of the particle it is likely to come to the surface and deteriorate the exterior of the particle. It is preferred to dry the granulated particles by hot air. The air should not be so hot as to melt or soften or decompose the granulated urea. Cooled air may then be passed over the particles until the residual moisture runs less than about 0.5%.

Example

A urea solution was concentrated at 100–125°C. for about 4 hours until its urea concentration was about 95%. The biuret content was then 3.9%. The heated and concentrated solution was sprayed at about 118°C. with about 100 mm Hg of pressure from a nozzle having an inside diameter of 0.8 mm. The granulation tower was 13 m high. Air at a temperature of 20°C. and a humidity of 60% was charged from the bottom of the tower at a speed of 0.8 m/sec.

The temperature of the particles on the bottom of the tower was about 75°C. The particles were immediately charged to a rotary kiln drier and countercurrently dried with hot air at a temperature of 60°C. at a speed of 2–3 m/sec. Cool air at 5°C. was then passed over the particles in the rotary kiln.

Distribution of particles size was as follows:

Over 8 mesh	12.2%
8–20 mesh	80.5%
Under 20 mesh	7.1%
Moisture content	0.4%
Hygroscopicity	60% of powder crystal urea.

What we claim is:

1. A method of producing storage-stable, spheroidal, granulated urea which comprises heating an aqueous solution of urea to a sufficient temperature to evaporate sufficient water therefrom to produce a 90–98% urea concentration in said solution as rapidly as possible without allowing the solution temperature to exceed 100°C. for more than five hours nor to exceed 80°C. for sufficient time to produce more than 5% biuret; then rapidly raising the solution temperature to from 115° to 130°C., if it is not already at that temperature, and spraying so-concentrated and heated solution into a gas-cooled granulation tower, whereby round discrete urea granules are obtained; collecting said granules; subjecting collected granules to the action of heated air at a temperature below the softening temperature of said granules until the residual moisture content does not exceed about 0.5% and cooling resultant granules to atmospheric temperature.

2. A method according to claim 1 wherein said evaporation is carried out at a temperature range of about 100–125°C. within a period of about four hours.

3. A method according to claim 1 wherein said granulation tower is gas-cooled by using air fed at about atmospheric temperature as the cooling and drying medium.

4. A method according to claim 1 wherein the said heated air is maintained at an average temperature of about 60°C.

5. A method of producing storage-stable, spheroidal, granulated urea substantially as

hereinbefore described with reference to the Example.

6. Granulated urea whenever produced in accordance with the method of any of the preceding claims.

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